Defense Threat Reduction Agency
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High Energy-Density Materials with Fast Energy Release: Molecular Scale Shock Response of Explosives

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TECHNICAL REPORT

May 2013

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14. ABSTRACT

This final report details the results of experiments designed to answer questions fundamental to shock initiation of explosives: (1) are electronic excitations prevalent in shock initiation? (2) does vibrational non-equilibrium contribute, and if so, which model is correct? (3) what chemical reactions occur, what are the kinetics, and what is the temperature that drives them? The answers to these questions underlie both the molecular level understanding of energetic material initiation and can justify or refute assumptions that must be made in modeling these processes. Femtosecond ultraviolet/visible absorption spectroscopy was used to address this issue of electronic excitation. Femtosecond stimulated Raman spectroscopy performed simultaneously in Stokes and anti-Stokes spectral regions was developed as a measurement of temperature and chemistry. Experimental results are presented for RDX (1,3,5-trinitroperhydro-1,3,5-triazine), a common molecular crystalline explosive, as well as for non-explosive materials.

15. SUBJECT TERMS

shock, explosives, spectroscopy, RDX

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CONVERSION TABLE

Conversion Factors for U.S. Customary to metric (SI) units of measurement.

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TO GET ◀──────────────── BY ◀──────────────── DIVIDE

angstrom	$1.000\ 000 \times E\ -10$	meters (m)
atmosphere (normal)	$1.013\ 25\ \times E\ +2$	kilo pascal (kPa)
bar	$1.000\ 000 \times E\ +2$	kilo pascal (kPa)
barn	$1.000\ 000 \times E\ -28$	meter^2 (m^2)
British thermal unit (thermochemical)	$1.054\ 350 \times E\ +3$	joule (J)
calorie (thermochemical)	$4.184\ 000$	joule (J)
cal (thermochemical/cm^2)	$4.184\ 000 \times E\ -2$	mega joule/m^2 (MJ/m^2)
curie	$3.700\ 000 \times E\ +1$	*giga bacquerel (GBq)
degree (angle)	$1.745\ 329 \times E\ -2$	radian (rad)
degree Fahrenheit	$t_k = (t°f + 459.67)/1.8$	degree kelvin (K)
electron volt	$1.602\ 19\ \times E\ -19$	joule (J)
erg	$1.000\ 000 \times E\ -7$	joule (J)
erg/second	$1.000\ 000 \times E\ -7$	watt (W)
foot	$3.048\ 000 \times E\ -1$	meter (m)
foot-pound-force	$1.355\ 818$	joule (J)
gallon (U.S. liquid)	$3.785\ 412 \times E\ -3$	meter^3 (m^3)
inch	$2.540\ 000 \times E\ -2$	meter (m)
jerk	$1.000\ 000 \times E\ +9$	joule (J)
joule/kilogram (J/kg) radiation dose absorbed	$1.000\ 000$	Gray (Gy)
kilotons	4.183	terajoules
kip (1000 lbf)	$4.448\ 222 \times E\ +3$	newton (N)
kip/inch^2 (ksi)	$6.894\ 757 \times E\ +3$	kilo pascal (kPa)
ktap	$1.000\ 000 \times E\ +2$	newton-second/m^2 (N-s/m^2)
micron	$1.000\ 000 \times E\ -6$	meter (m)
mil	$2.540\ 000 \times E\ -5$	meter (m)
mile (international)	$1.609\ 344 \times E\ +3$	meter (m)
ounce	$2.834\ 952 \times E\ -2$	kilogram (kg)
pound-force (lbs avoirdupois)	$4.448\ 222$	newton (N)
pound-force inch	$1.129\ 848 \times E\ -1$	newton-meter (N-m)
pound-force/inch	$1.751\ 268 \times E\ +2$	newton/meter (N/m)
pound-force/foot^2	$4.788\ 026 \times E\ -2$	kilo pascal (kPa)
pound-force/inch^2 (psi)	$6.894\ 757$	kilo pascal (kPa)
pound-mass (lbm avoirdupois)	$4.535\ 924 \times E\ -1$	kilogram (kg)
pound-mass-foot^2 (moment of inertia)	$4.214\ 011 \times E\ -2$	kilogram-meter^2 (kg-m^2)
pound-mass/foot^3	$1.601\ 846 \times E\ +1$	kilogram-meter^3 (kg/m^3)
rad (radiation dose absorbed)	$1.000\ 000 \times E\ -2$	**Gray (Gy)
roentgen	$2.579\ 760 \times E\ -4$	coulomb/kilogram (C/kg)
shake	$1.000\ 000 \times E\ -8$	second (s)
slug	$1.459\ 390 \times E\ +1$	kilogram (kg)
torr (mm Hg, 0^0 C)	$1.333\ 22\ \times E\ -1$	kilo pascal (kPa)

*The bacquerel (Bq) is the SI unit of radioactivity; 1 Bq = 1 event/s.

**The Gray (GY) is the SI unit of absorbed radiation.

EXECUTIVE SUMMARY

The principle goal of this $150K/yr, 3-yr. project was to experimentally measure the time evolution of shock induced chemistry in the explosive RDX. Since direct measurements of shock induced chemistry in RDX, or any other explosive, have eluded researchers for several decades, new techniques had to be developed. Transient absorption was coupled to laser shock loading to probe the possible role of electronic excitations in the chemistry of RDX. Transient absorption signatures turned out to be a good indication of the relative degree of chemical reaction versus time, but were not conclusive regarding whether electronic excitation was an important factor in shock initiation. Preliminary efforts to sensitize shock induced chemistry with ultraviolet pulsed excitation were performed, but are not yet conclusive. The bulk of the effort of the project was directed at development of femtosecond stimulated Raman spectroscopy (FSRS) as a diagnostic of temperature in the presence of chemistry. This effort was significantly more difficult than initially anticipated. While we had planned to apply the method to single shot laser experiments, the sensitivity achieved during the project duration was insufficient to measure the sub-nanogram amounts of material shocked. Despite the difficulties applying this measurement to our current small scale laser shock experiments, it shows significant promise for measurements where there is more volume of material shocked. We have established the theoretical framework and experimentally validated FSRS as a sub-picosecond molecular vibrational temperature measurement diagnostic. We have also followed the shock induced chemistry of RDX through transient absorption spectroscopy and established the shock stresses required to achieve sub-nanosecond reaction rates.

The project led to 3 peer reviewed journal articles, 4 conference proceedings, 3 invited talks at conferences, 2 contributed talks at conferences, and a number of other presentations.

INTRODUCTION

A. RESEARCH GOALS

The initial events that occur upon shock loading explosives are crucial in determining the sensitivity of the material towards accidental detonation. While various theories have been proposed regarding the shock induced reaction mechanisms, there are few experiments sensitive to the time and length scales involved in testing them.

The goals of this project were to experimentally address these fundamental underpinnings of explosive initiation and detonation physics. We sought to:

> 1) determine the extent to which electronic excitations are, or are not, involved in shock induced reactions. Measurement of potential sensitization of explosives by electronic excitation was a secondary goal.

> 2) measure the mechanism(s) of shock induced vibrational excitation of chemistry in explosives, particularly testing the multiphonon up-pumping hypothesis.

3) provide data of unprecedented detail on the initial evolution of temperature and chemistry following the shock loading of explosives on scales amenable to comparison to molecular dynamics simulations.

Completing these goals is a prerequisite to identification of the proper theoretical treatment of shock induced initiation at the molecular level. This is essential for predictive capability regarding how current explosives will function in untested conditions and for development and optimization of future explosive materials.

B. BACKGROUND.

What we don't know, and why we don't know it – what happened just before the experiment blew up

Shock and detonation physics is a field central to the missions of the Department of Energy (DOE) and the Department of Defense (DoD). Huge investments have been made and continue to be made into establishing the capability to *predict* how explosives will react in any given scenario. Yet on the molecular level, very little is known about the dynamic response of these materials. Detonation requires the energy of chemical reactions to sustain it. How does energy from an impact couple into molecules? What is the chain of chemical reactions induced? How fast do they occur? If the mechanism is thermal, what is the temperature that drives the reactions?

Why don't we already have the answers to these important questions? Traditional explosively driven or gas gun impact experiments have formed the foundations for shock and detonation physics, but spectroscopic monitoring of bulk (mm-cm) scale shocked samples at nanosecond time scales does not allow resolution of chemistry. This is due to both shock induced sample opacity (due to the sample thickness) and subnanosecond reaction rates. A handful of large scale experiments have utilized Stokes/anti-Stokes Raman scattering to measure temperature at nanosecond time scales, but the inherent complexity and expensive nature of gas gun experiments has prohibited more than a few experiments. Typically, the key parameter of temperature is not measured at all.

C. RELEVANCE.

Impact on understanding and modeling of explosive initiation

With no direct experimental data constraining models of shock induced reactions on the molecular scale, it is unclear which underlying assumptions are appropriate. The most advanced reactive molecular dynamics simulations (such as those using the force field REAX) offer tremendous potential insight into the molecular level shock response that underpins detonation. However, these, and all other, classical molecular dynamic simulations assume that only the electronic ground state is involved. If electronic excited states are present, the results of these simulations could not be expected to be correct. In opposition, solid state quantum calculations have been used to suggest that electronic excitations are essential to explosive initiation. Which is correct? Is the vibrational energy flow in thermal equilibrium, or does reaction occur before the energy is thermalized? We need to measure the transient vibrational and electronic absorption

spectra of shocked energetics during initiation to directly and definitively answer these questions.

Relevance to DTRA Missions

The application, optimization, and therefore fundamental understanding of explosives is essential to DTRA missions. Initiation of explosives occurs through a chain reaction, where localized molecular insults lead to the collective emergence of the phenomena of detonation. Understanding the initial events is crucial in predicting, and potentially tailoring shock sensitivity of explosive materials. This work is relevant to the long term objectives of the DTRA Basic Research to Counter Weapons of Mass Destruction, Thrust 4, Topic E: "Basic science to defeat WMD involves furthering the understanding of explosives, their detonation and problems associated with accessing the target WMDs."

METHODS

Prior to this project, we have developed methods to generate and characterize gas gun-like shocks (up to 60 GPa, 300 ps constant pressure) on very small scales – (100 μm diameter x 0-2 microns thickness) with femtosecond synchronization to spectroscopic probing. Figure 1 illustrates the spatial and temporal characteristics of the drive laser and the diagnostics employed. While the ellipsometry diagnostic was well developed, the transient absorption (TA), femtosecond stimulated Raman spectroscopy (FSRS) and coherent anti-Stokes Raman scattering (CARS) diagnostics were under development during the project. Sample materials studied in this project included single crystal
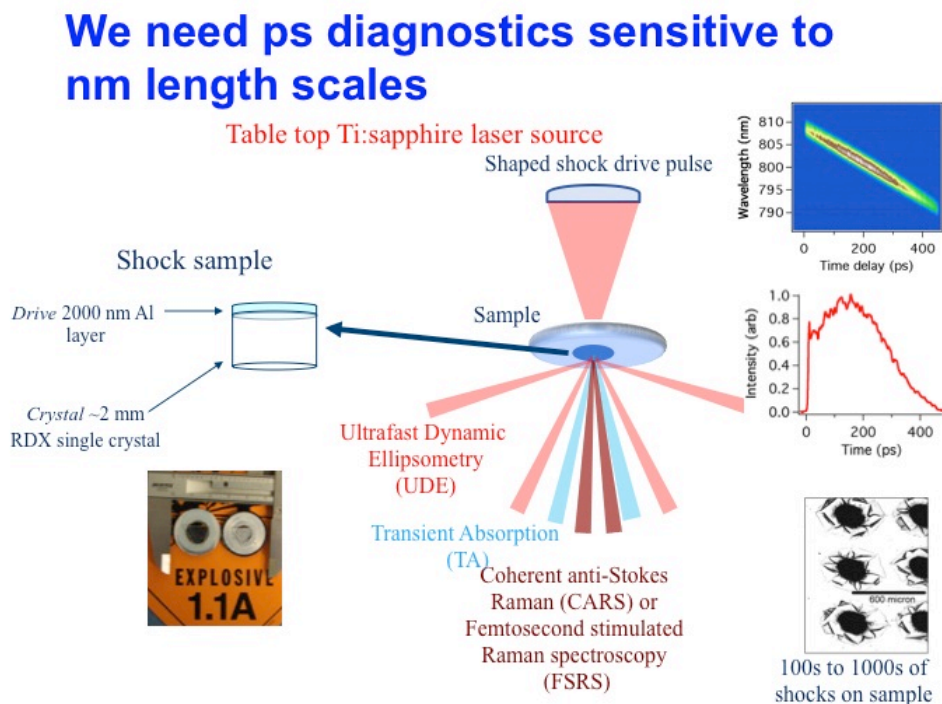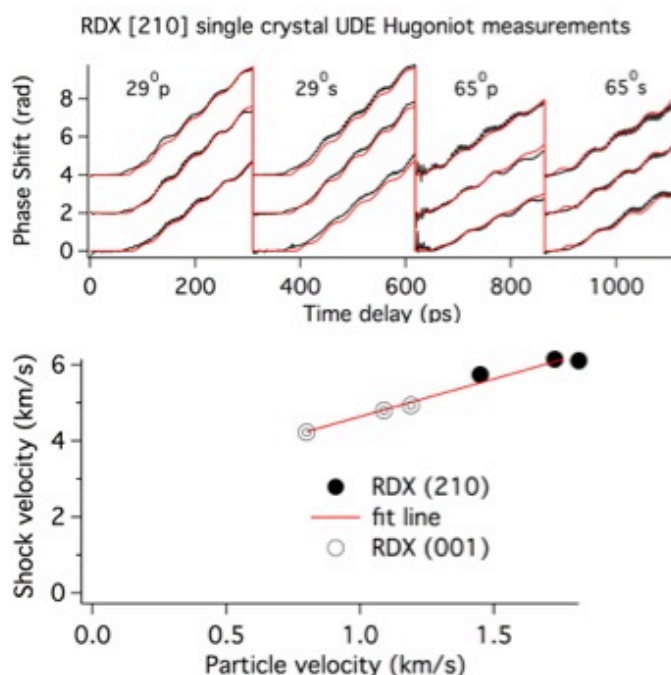

Figure 1 Schematic of experimental methods detailing shock size and duration as well as typical sample geometries used in the laser shock experiments.

explosives, drop cast films of explosives, and inert (nonexplosive) materials for reference and diagnostic testing.

RESULTS

A. ULTRAFAST DYNAMIC ELLIPSOMETRY (UDE)

Careful shock physics experiments require a well defined shock state. We measure the shock state interferometrically using UDE in order to define the shock and particle velocity experienced by the material. These are the most commonly measured experimental parameters, and allow us to compare our results to those of larger scale explosive or gas gun driven experiments. The UDE measurements set the stage for the advanced spectroscopic diagnostics that will provide information about the material response at the molecular level.

Figure 2 illustrates some of the data acquired on single crystal RDX, with UDE measured over ~ 300 ps at two angles of incidence and two polarizations. The upper diagram of Fig. 2 shows the experimental measurements for three shots and the theoretical fits. The shock states derived from the fits are shown in the shock velocity/particle velocity plane in the lower Figure. For RDX single crystals, there is no published Hugoniot for us to compare these data to. Algorithmic advances in the data analysis have allowed us to determine Hugoniot data with increased accuracy and expediency that is expected to lead to publication of the RDX single crystal Hugoniot once more analysis is complete.

Figure 2 UDE measurements of shocked RDX (black curves, top) fit to theory (red curves, top) allow the determination of Hugoniot parameters that define the shock state (bottom).
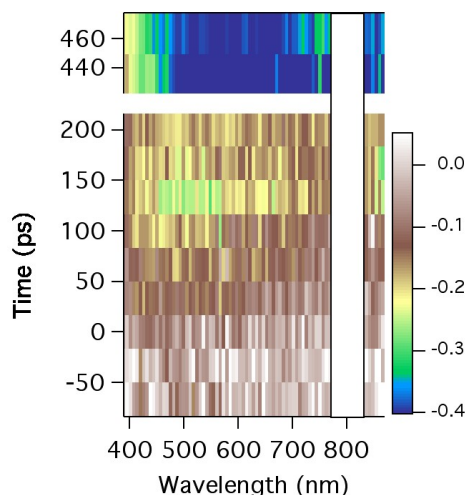
B. TRANSIENT ABSORPTION

The transient absorption of RDX was studied as a function of input shock stress for single crystals and drop cast films. For shocks near or above ~ 20 GPa, substantial new absorptions appeared within 300 ps of shock loading. Figure 3 shows results for a series of single crystal experiments. Figure 4 shows analogous results for shock loading of drop cast films of RDX. The drop cast films exhibit faster reaction rates, presumably due to the increased heterogeneity of the nano-microcrystals compared to the millimeter scale single crystal of Fig. 3. Heterogeneity can lead to hot spot formation and increased reaction rates at boundaries. The results shown in Figures 3 and 4 are the most direct data
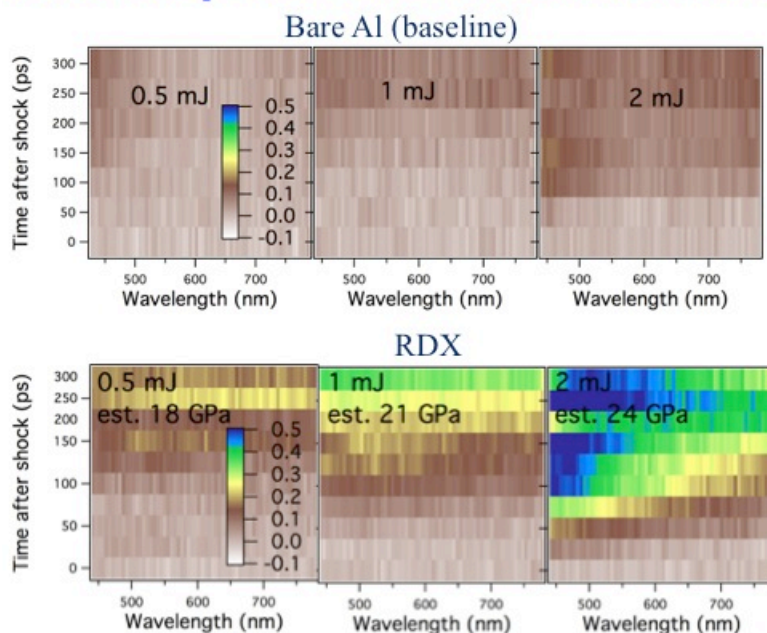
Figure 4 Loss of reflectivity due to the buildup of chemical products is apparent in the transient absorption data of RDX single crystal shocked to ~23 GPa.


Figure 3 Drop cast RDX films exhibit strong transient absorption features attributed to chemical reaction product formation that is a strong function of input shock strength.

available indicating reaction rates on subnanosecond time scales in strongly shocked explosive materials and the shock stress required to achieve these rates.

A question arose regarding the interpretation of the transient absorption data under the potential influence of scattering due to shock microfracturing. Scattering would also lead to a reflectivity loss, but not indicate chemical reaction. To address this possibility, we added a probe that measured the scattering of the transient absorption probe. The time dependent scattering intensity was measured following the shock, and shown to be a significant but small fraction of the transient absorption observed. These data are shown in Fig. 5.

While the transient absorption results clearly indicated chemical reaction leading to
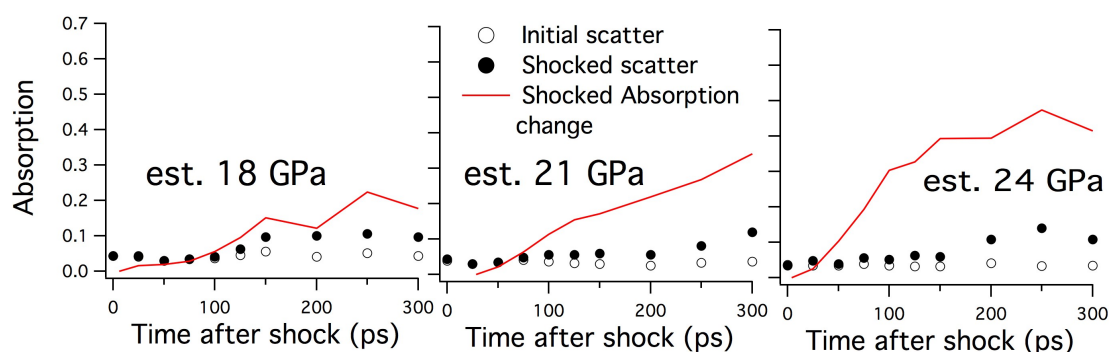
Figure 5 Time dependent spectrally integrated scattering of the transient absorption probe was measured. Scattering was a small but measurable part of the loss of reflectivity. Scattering increased with shock strength and time after shock, but increased slower than the transient absorption due to chemical product formation.

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product absorption in the visible spectral region, the peaks are too broad and featureless to provide information on the molecular structure of the reaction products. Time resolved vibrational spectroscopies such as FSRS or CARS are more appropriate for answering questions of detailed chemical kinetics.

C. FEMTOSECOND STIMULATED RAMAN SPECTROSCOPY FOR TEMPERATURE MEASUREMENT, FSRS(T)

FSRS is a vibrational spectroscopy that can be used both for determination of chemical species and nonequilibrium vibrational temperatures following shock loading. The initial goal was to use a Stokes/anti-Stokes ratio of FSRS peaks to measure the transient energy
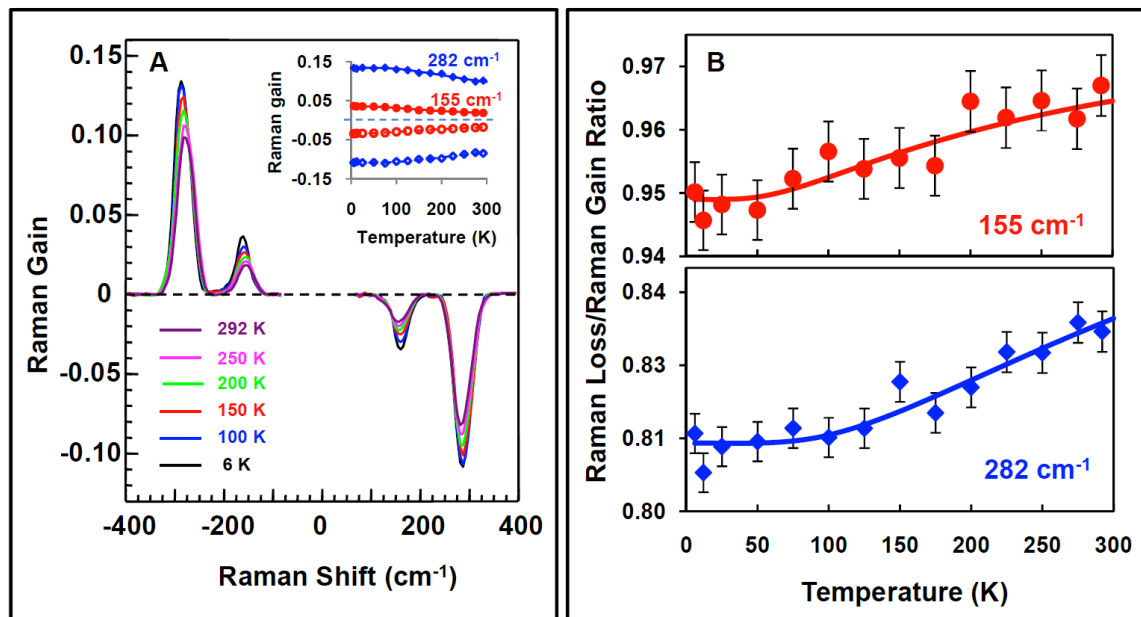

Figure 6 The temperature dependence of FSRS Stokes to anti-Stokes peaks was measured as a function of temperature and shown to match the new theoretical predictions.

flow in shocked molecules. Initial experiments and theoretical probing led to the conclusion that FSRS(T) had a different Stokes/anti-Stokes temperature dependence than spontaneous Raman. We developed both the theoretical underpinnings of the temperature dependence and validated these predictions experimentally with static experiments. The results of these static experiments are summarized in Figure 6. The effect of temperature on the Stokes to anti-Stokes FSRS ratio was small but measurable. The FSRS signals themselves were much easier to measure than the accurate ratio of intensities on the Raman gain (Stokes) and Raman loss (anti-Stokes) side. We established the equations for the temperature dependence, and also the temperature independence of the Stokes to anti-Stokes signals for other common nonlinear vibrational spectroscopies such as CARS or the Raman induced Kerr effect (RIKES). Details of these advances can be found in the resulting publications.
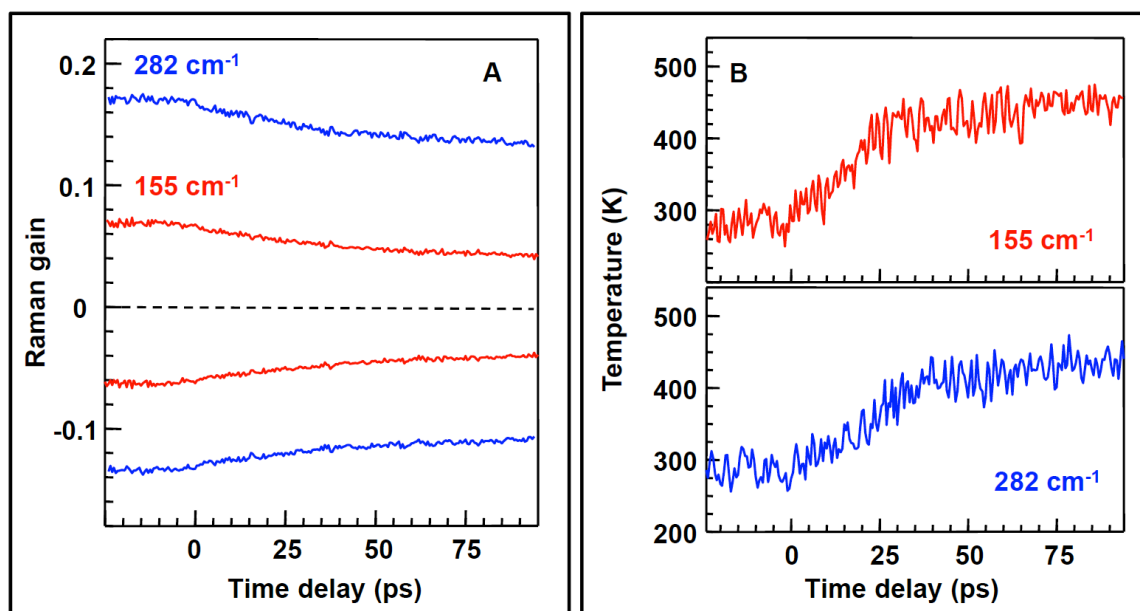
Figure 7 The transient temperature dynamics (B) of photoexcited calcite crystal was monitored as a function of time for two different low frequency modes through their FSRS signals (A).
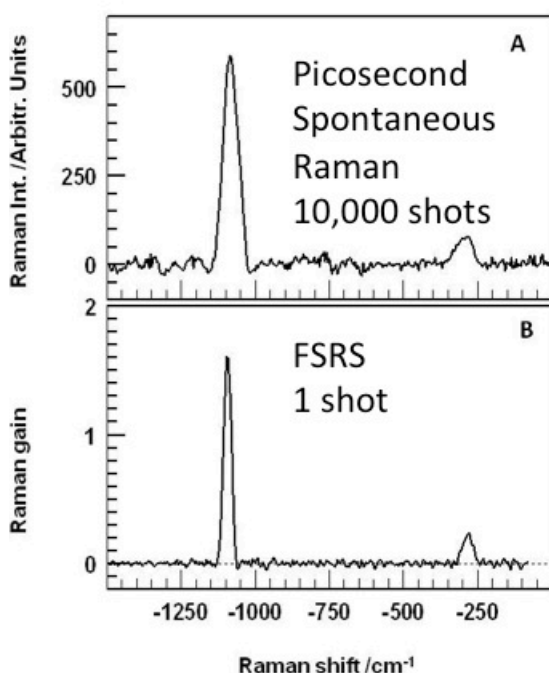
Figure 8 Sensitivity of picosecond spontaneous Raman versus FSRS under identical conditions

Following the static studies establishing the validity of FSRS(T), we utilized it to measure transient temperature dynamics. We performed multiphoton electronic excitation of calcite crystals as an initial experiment. Figure 7 shows the 30 ps temperature rise of the low frequency vibrational modes following femtosecond photoexcitation. The measurement response time was sub-picosecond, but 30 ps was required for the electronic energy to thermalize into the vibrational modes probed. The capability of FSRS(T) to measure picosecond vibrational temperature dynamics was established through the data shown in Figure 7.

Following the validation experiments, there were two clear requirements to transition FSRS(T) to shock experiments. First, the method should be implemented in a single shot configuration sensitive to small amounts of material. Second, the effects of chemistry and temperature must be deconvolved.

After changing the experimental configuration to allow single shot FSRS acquisition, we determined the sensitivity of FSRS compared to spontaneous Raman spectroscopy. FSRS

signals could be acquired >90,000 x faster than picosecond spontaneous Raman under the exact same excitation and collection conditions. Data from 10,000 shots of picosecond spontaneous Raman are compared to a single shot FSRS spectrum in Figure 8. Further theoretical analyses also allowed the separation of simultaneous changes in chemistry and temperature by using multiple vibrational peaks. Details of the experimental and theoretical results have been submitted for publication in the Journal of Raman Spectroscopy. These analyses will be crucial for utilization of FSRS(T) under shock conditions. While we were unable to measure FSRS on our shocked samples due to the small amount of material (~100 ng), larger laser shock experimental facilities may benefit from the foundational work performed on this technique under this project.

D. TRANSIENT ABSORPTION WITH ULTRAVIOLET ELECTRONIC EXCITATION

If there are electronic states participating in the shock induced chemistry of detonations, electronic excitation *prior* to shock loading may seed or sensitize the shock induced chemistry. To study this possibility, we added a 400 nm multiphoton excitation to the sample side of our shock experiment. A thin film RDX sample was photo-excited simultaneous to the shock arrival and probed 200 ps after with transient absorption spectroscopy. Shock sensitization would be expected to lead to more product formation and larger transient absorption. The preliminary experiments showed the opposite effect, with substantially less transient absorption apparent. Further studies to isolate possible interfering phenomena have not yet been performed. Since a great deal of time was spent developing the spectroscopic diagnostics, only preliminary results on the effect of excitation could be attained under the scope and timeline of this project.

CONCLUSIONS

The UDE experimental results have provided accurate Hugoniot data for RDX single crystals that are not currently available. The transient absorption and transient scattering experiments produced the first data to show relative reaction rates on sub-nanosecond time scales as a function of shock stress. These data will be valuable for validation of reactive theoretical models in the future. FSRS(T) was established as a sub-picosecond molecular thermometric probe, both theoretically and experimentally. While the initial very ambitious goals of the project were only partially met, the project has claimed success on several fronts advancing efforts in shock induced chemical problems relevant to DTRA's long term mission.

PROGRAMMATIC INFORMATION

Project scope:

The project was performed over the time period July 2009-Sept. 2012. The total funding of the project was $450K.

Personnel supported:

Primary personnel support was for a postdoctoral researcher (Nhan Dang). The PI was also partially supported (~10%) by the project. A summer undergraduate student (Robert Chalmers, Whitworth College) was partially supported in 2009. A small amount of funds also supported a technician (Virginia Hamilton) to polish RDX crystals.

METRICS

A. PUBLICATIONS

"Molecular scale shock response of explosives: electronic absorption spectroscopy", S. D. McGrane, D. S. Moore, V. H. Whitley, C. A. Bolme, and D. E. Eakins, *Shock Compression of Condensed Matter AIP Conf. Proc.* (2009)

"Transient absorption spectroscopy of laser shocked explosives", S. D. McGrane, N. C. Dang, V. H. Whitley, C. A. Bolme, and D. S. Moore , *International Detonation Symposium Proceedings* (2010)

"Ultrafast dynamic ellipsometry and spectroscopy of laser shocked materials", S. D. McGrane, C. A. Bolme, V. H. Whitley, and D. S. Moore, *High Power Laser Ablation Conference Proceedings*, (2010)

"Ultrafast dynamic ellipsometry and spectroscopies of laser shocked materials", S. D. McGrane, C. A. Bolme, V. H. Whitley, and D. S. Moore, *Ultrafast Phenomena Conference*, (2010)

"Femtosecond Stimulated Raman Scattering Picosecond Molecular Thermometry in Condensed Phases", N. C. Dang, C. A. Bolme, D. S. Moore, and S. D. McGrane, *Phys. Rev. Lett.*, 107, 43001 (2011)

"Shock Induced Chemistry in Liquids Studied with Ultrafast Dynamic Ellipsometry and Visible Transient Absorption Spectroscopy", Nhan C. Dang, C.A. Bolme, David S. Moore, and Shawn D. McGrane, just accepted by *J. Phys. Chem. A* on web at http://pubs.acs.org/doi/pdf/10.1021/jp307464w

"Temperature measurements in condensed phases using non-resonant femtosecond stimulated Raman scattering", N. C. Dang, C. A. Bolme, D. S. Moore and S. D. McGrane, in review *J. Raman Spec.*

B. PRESENTATIONS

Invited talks:

•2010 High Power Laser Ablation Conference, "Ultrafast dynamic ellipsometry and spectroscopy of laser shocked materials", presented by S. D. McGrane

•2010 Gordon Research Conference on Energetic Materials, "Ultrafast shock response in energetic materials", presented by S. D. McGrane

2011 Shock Compression of Condensed Matter, "Shocked reactions: the first half nanosecond", presented by S. D. McGrane

Contributed talks:

•2010 International Detonation Symposium- "Transient absorption spectroscopy of laser shocked explosives", presented by S. D. McGrane

2012 LANL Advanced Thermometry Workshop, "Femtosecond stimulated Raman picosecond molecular thermometry", presented by S. D. McGrane

2011 Shock Compression of Condensed Matter - "Temperature measurement in condensed phases using femtosecond stimulated Raman scattering", presented by Nhan Dang

Poster presentations:

2010 LANL Chemistry Capabilities Review, "Ultrafast shock physics and chemistry", presented by S. D. McGrane

2010 LANL Postdoc Research Symposium, "Towards a Universal Picosecond Molecular Thermometer", presented by Nhan Dang

2010 Air Force Office of Scientific Research Meeting on Chemical Dynamics, "Ultrafast shock physics and chemistry", presented by S. D. McGrane

2010 Int. Conf. on Ultrafast Phenomena, "Ultrafast dynamic ellipsometry and spectroscopy of laser shocked materials", presented by S. D. McGrane

2012 Chemistry in Dynamic Extremes Workshop, "Ultrafast shock chemistry measurement development", presented by N. C. Dang

C. INTERACTIONS/TRANSITIONS

Collaborations and interactions initiated during the project

Profs. I. Oleynik and V. Zhakhovsky at Univ. South Florida (MD simulations of shock processes)

Alex Tappan of Sandia National Laboratory is providing PETN thin films with potential nano-RDX films in the future

Laboratory visits by international scientists:

Dr. Phillipe Herbert (CEA, France) spent 1 week at our lab learning our methods to couple our experiments to his.

Prof. T. Sano (Osaka), N. Ozaki (Osaka), Dr. Sekine (Nat. Inst. For Mat. Sci., Tsukuba, Japan) visited and continue shock physics collaborations on non-energetic materials.

Drs. Sean Kearney, Darcie Farrow, Brook Jilek, Ian Kohl and Junji Urayamaof Sandia National Laboratory are now starting to build an ultrafast laser shock capability

Drs. Mike Armstrong, Jonathan Crowhurst, and Joe Zaug of Lawrence Livermore National Laboratory are using ultrafast laser shocks to study energetic and other material dynamics

D. OTHER

This work has not yet transitioned out of basic research.

New discoveries, inventions, or patent disclosures

No new marketable discoveries, inventions, or patents have been produced by this work.

Honors/Awards:

No new honors or awards have been produced by this work.

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